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CHARACTERIZATION AND ANALYSIS OF DEVONIAN SHALES AS RELATED TO RELEASE OF GASEOUS HYDROCARBONS

Quarterly Technical Progress Report, September—November 1976

M. J. Snyder
J. R. Schorr

December 1, 1976

Work Performed Under Contract No. E(40-1)-5205

Battelle Columbus Laboratories
Columbus, Ohio

ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION

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September-November

M. Jack Snyder and J. R. Schorr

BATTELLE
Columbus Laboratories
505 King Avenue
Columbus, Ohio 43201

Date Published-December 1, 1976

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INTRODUCTION

This program which is a part of The Resource Inventory and Shale Characterization subprojects of ERDA's Eastern Shale Project, was initiated in September 1976 with an effective contract period of July 1, 1976 through September 30, 1977. During July and August, in anticipation of the execution of the contract, work was started in connection with the sampling of cores from Devonian shale wells which had been or were being drilled and cored. Active laboratory work was started in September. This first quarterly technical progress report covers progress made in the core sampling work and the laboratory characterization work during the quarter from September through November, 1976.

OBJECTIVE AND SCOPE

The objective of this program is to determine the relationships between shale characteristics, hydrocarbon gas content, and well location to provide a sound basis for defining the productive capacity of the Eastern Devonian shale deposits and for guiding research, development, and demonstration projects to enhance the recovery of natural gas from the shale deposits. The program includes a number of elemental tasks as a part of the Resource Inventory and Shale Characterization subprojects of ERDA's Eastern Shale Project and is designed to provide large quantities of support data for that project.

Approximately 100 core samples of gas bearing Eastern Devonian shale will be examined in the program. After the characterization data for individual wells have been compiled, a regression-type analysis for

pattern recognition will be performed to establish the interrelationship between the shale characteristics, the hydrocarbon gas content, and well locations from which the samples were obtained.

The work during the first 18-month period of this program comprises six tasks:

Task	Descriptive Title
1	Core Sampling
2	Gas Content and Gas Release Kinetics
3	Chemical Characterization of Shale
4	Physical Characterization of Shale
5	Lithology of Shale
6	Data Interpretation and Correlation

Task 1. Core Sampling

In cooperation with representatives of ERDA and of the cognizant State Geological Survey or The U.S. Geological Survey, (1) participate in the selection of core samples for characterization in the Battelle program, and (2) encapsulate the selected samples in special sealed containers to preserve their approximate "down-hole" condition and gather relevant information from on-site observations and well-log data for subsequent interpretation and correlation with characterization results on the selected samples.

Task 2. Gas Content and Gas Release Kinetics

Determine the composition of and quantity of hydrocarbon gas evolved from and surrounding the shale samples in the sealed containers. Determine the gas release kinetics by observations of the rate of pressure build-up in the sealed containers after initial release of pressure for selected samples having high initial pressures and by thermal gravimetric measurement of weight loss as a function of time at three different temperatures for specimens cut from the samples after removal from the sealed containers.

Task 3. Chemical Characterization of Shale

Determine total carbon, hydrogen, and nitrogen contents and, where appropriate, total carbonate content for all samples. Determine trace elements by optical emission spectroscopy on selected samples from each well.

Task 4. Physical Characterization of Shale

Characterize the microstructure of the samples in terms of porosity (total, open, and closed), pore size distribution, total surface area, and gas permeability.

Task 5. Lithology of Shale

Determine the mineralogical composition of the samples by x-ray diffraction techniques and define the morphology of the minerals and their relationship to the organic debris by thin-section petrography. Ascertain structural changes resulting from heating by observations on duplicate samples from the same core sample. Examine selected samples from each well by scanning electron microscopy to supplement the petrographic observations.

Task 6. Data Interpretation and Correlation

Compile the data from the field observations, well logs, and laboratory characterizations and observations into a data package for each well for input to the regression-type analysis, for input to the USGS data bank, and for transmission to the ERDA technical project officer. Determine, using a pattern recognition statistical analysis technique and conventional data interpretation methods, the interrelationships and trends between the hydrocarbon gas content at various levels, the well head observation, and the lithological, chemical, and physical characteristics of the shale for individual wells and as a function of location.

SUMMARY OF PROGRESS

Progress on each of the tasks during the period covered by this report is compared with the plan in Figure 1. A milestone chart showing progress on the various tasks for each of the wells sampled is shown in Figure 2. Major effort was devoted to Task 1 (Core Sampling) during the period; sample collection has been more rapid and intensive than was anticipated. A total of 265 sealed core samples were acquired during this period. An initial selection of 10 samples was made based on the available well-log data for two of the wells and the analysis of those samples for initial gas content and initial release characteristics was started in September. Effort on Tasks 3, 4, and 5 was was initiated late in September. Task 6 efforts on data interpretation and correlation started late in November, but insufficent data are available yet to permit meaningful interpretation. A total of 56 samples are now somewhere along the characterization chain and an additional 20 to 25 samples are being started each month.

DETAILED DESCRIPTION OF TECHNICAL PROGRESS

Task 1. Core Sampling

Sealed core samples from six different wells were acquired during July through November, 1976. The samples from the first two wells were selected and encapsulated by representatives of Columbia Gas System Service Corporation in January 1976 and were supplied to Battelle's Columbus Laboratories in August 1976 for use in the program. A Battelle technical staff member, Mrs. Diane Hooie, was the Battelle representative on the sample selection team for the other four wells and was responsible for on-site observations, the encapsulation of the selected samples, and their transportation to the Battelle-Columbus Laboratories. Sealed samples from three of the wells were also prepared by Mrs. Hooie for use by other ERDA contractors. Mr. Alvin J. Walker, another Battelle technical

ERDA CONTRACT E(40-1)-5205

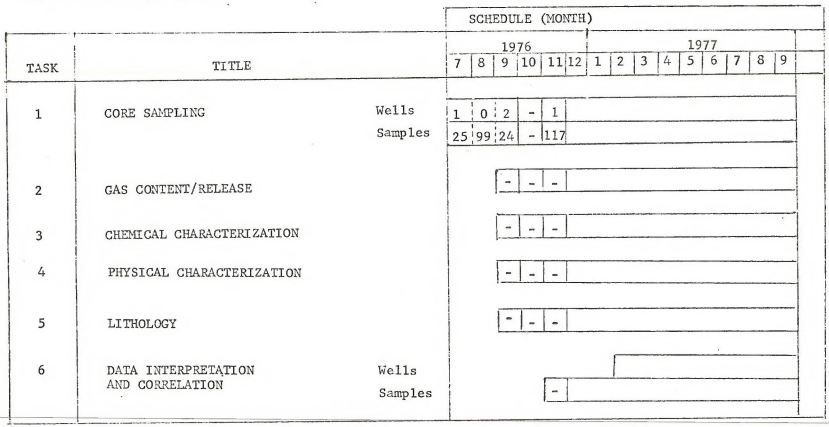


FIGURE 1. PROGRAM PLAN AND PROGRESS

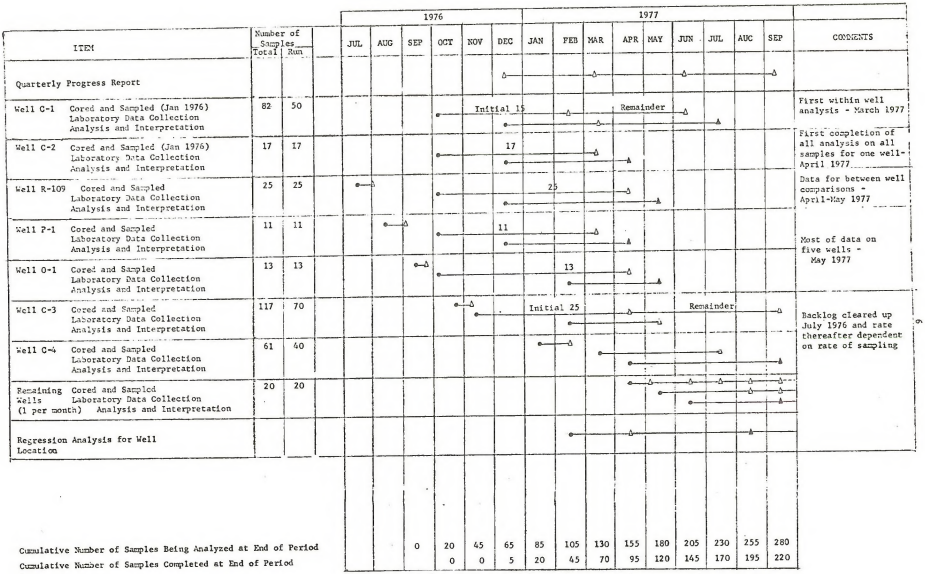


FIGURE 2. MILESTONE CHART

staff member on the project, assisted in the selection and encapsulation of some of the samples from Well No. C-366 and will act as an alternate to Mrs. Hooie in the future. Pertinent information about the location of the wells, the depth interval cored, and the number and disposition of sealed samples is given in Table 1.

A copy of the worksheet used to record on-site observations about each sample prior to canning is shown in Figure 3.

Data from well-logs at the depths corresponding to each of the samples selected were recorded on punched cards for subsequent data analysis. Computer-generated plots of the data were used to guide selection of the initial set of samples for analysis. A plot of the neutron porosity data versus well depth for Well C-1 is shown in Figure 4. Seven samples were selected from Well C-1 and three samples were selected from Well C-2 for the initial set of samples for characterization. Samples have now been selected from each of the wells, except the most recent one (C-336), and are now being characterized.

Task 2. Gas Content and Gas Release Kinetics

The can sampler illustrated in Figure 5 was used in determining the pressure and volume of the gas surrounding the shale samples and to transfer the gas from the sealed can to the gas analysis system. The initial pressure, volume, and composition data have been gathered on selected samples from each of the wells except the most recent one (Well C-336). Figure 6 is a copy of a data sheet from one of the initial gas content measurements.

The data have been entered on punched cards and the relationships among the gas release data and the well log data as a function of depth are being examined. Typical computer-generated plots illustrating some of the comparisons are given in Figures 7, 8, and 9. As information becomes available from the other tasks on the physical and chemical characteristics of the cores, their relationships with one another and with the gas content data will be analyzed.

TABLE 1. IDENTIFICATION OF CORED WELLS AND SAMPLES SELECTED

Well Code Number	Location of Well	Well Operator	Date Sampled	Core Depth Interval, Feet	Number of Samples
C-1	Lincoln County, WV	Columbia Gas	Jan. 1976	2746 - 4045	82
C-2	Lincoln County, WV	Columbia Gas	Jan. 1976	2655 - 3971	17
R-109	Washington County, OH	River Gas	Jul. 30, 1976	3494 - 3705	25
P-1	Sullivan County, IN	Energy Resources of Indiana, Inc.	Sept. 1, 1976	2495 - 2595	11[a]
0-1	Christian County, KY	Orbit Gas	Sept. 18, 1976	2183 - 2319	13[b]
C-336	Martin County, KY	Columbia Gas	November 6, 1976	2434-3405	117[c]

(a) An additional 6 samples were selected and encapsulated for other ERDA contractors.

(b) An additional 4 samples were selected and encapsulated for other ERDA contractors.

(c) An additional 90 samples were selected and encapsulated for other ERDA contractors.

SAMPLE NO. C-246-D621
depth 0.15
0.35

(1) Rock color N-5

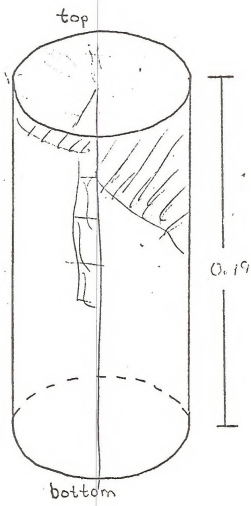
(2) type shale Devonian

(3) shale & other minerals detected
pyrite, siderite
(?)

(4) no. of sample pieces 2

(5) gas detected small top
oil detected none

(6) Fracture description see
details mentioned here & will be
given in written form
(?)



1750

time out of hole 3 hrs 5 min
date out of hole 11 Dec 70
engineer D. V. Hess

FIGURE 3. ON-SITE DESCRIPTION

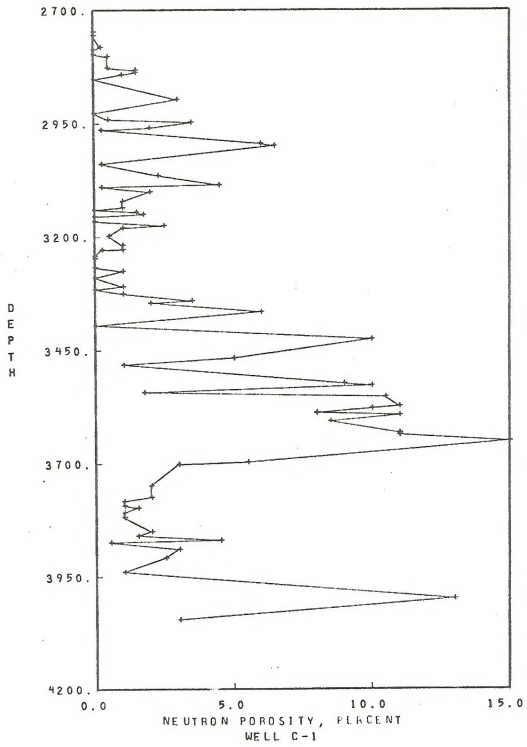


FIGURE 4. PLOT OF WELL LOG DATA FOR SAMPLES FROM C-1

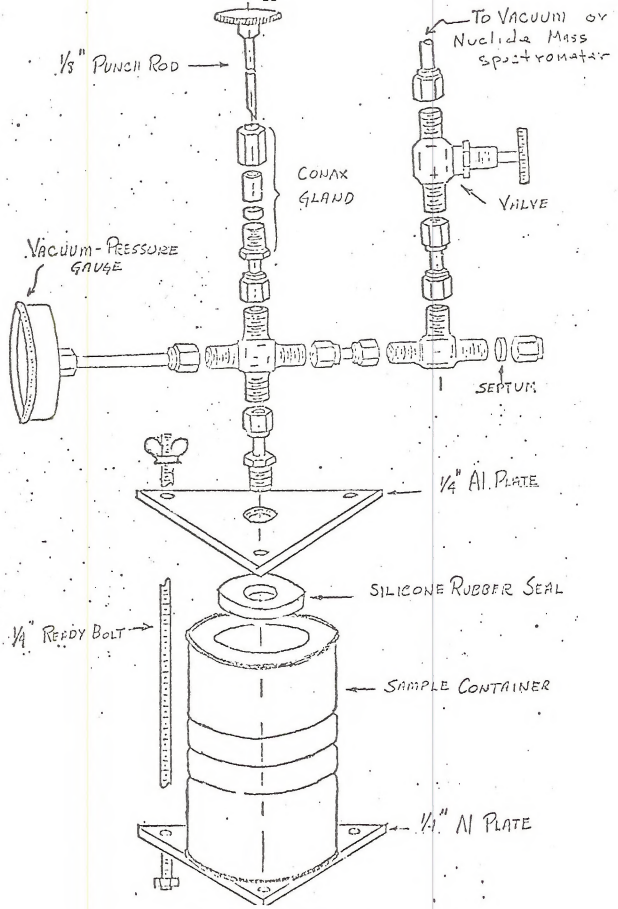


FIGURE 5. B.C.L. CAN SAMPLER

SAMPLE NO. C-1-3701

FIGURE 6. DATA SHEET - OFF GAS MEASUREMENTS

Initial

Weight of core and can: _____ g

Weight of can: _____ g

Weight of core: _____ g

Date received for off-gas measurements: 11/4/76

Punctured date: 11-10-76 time: 11:20 A.M.

Initial gage reading: 1060 Torr

Composition of off-gas (volume %):

				% of total hydrocarbon
N_2	12.17	C_1	58.65	69.5
O_2	1.97	C_2	15.81	18.7
CO	—	C_3	7.87	9.3
CO_2	1.41	C_4	1.74	2.1
		C_5	0.37	0.4
			84.44	

NPS

Pressure rebuild data (optional):

time after initial off gas (days) _____ _____ _____

pressure (Torr) _____ _____ _____

Comments: CAN RUSTED TOP TO BOTTOM ~ ½" WIDE 11-4-76

CAN BULGED 11-4-76

Work Completed by: Melanie E Davies

Date returned to CMS: 11/11/76 Received by Chuck

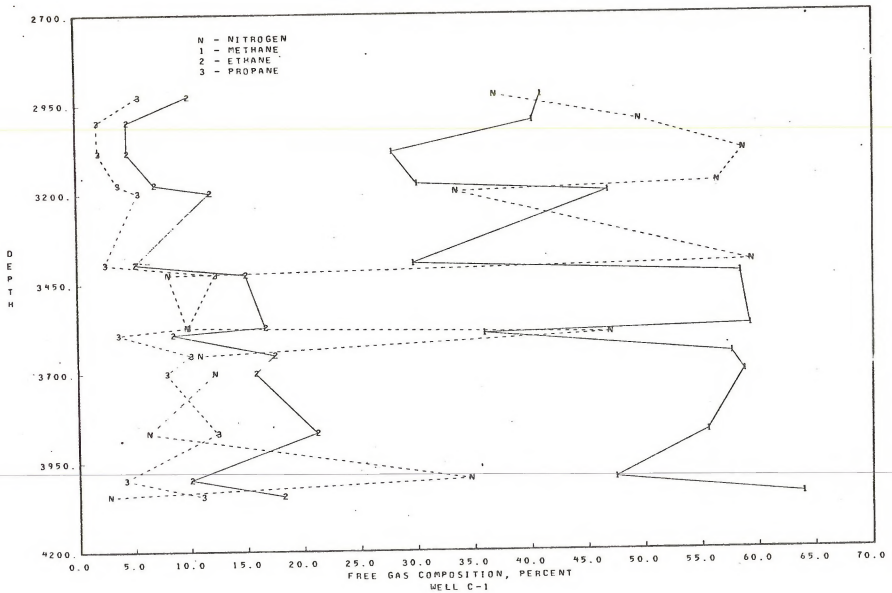


FIGURE 7. COMPOSITION OF FREE GAS

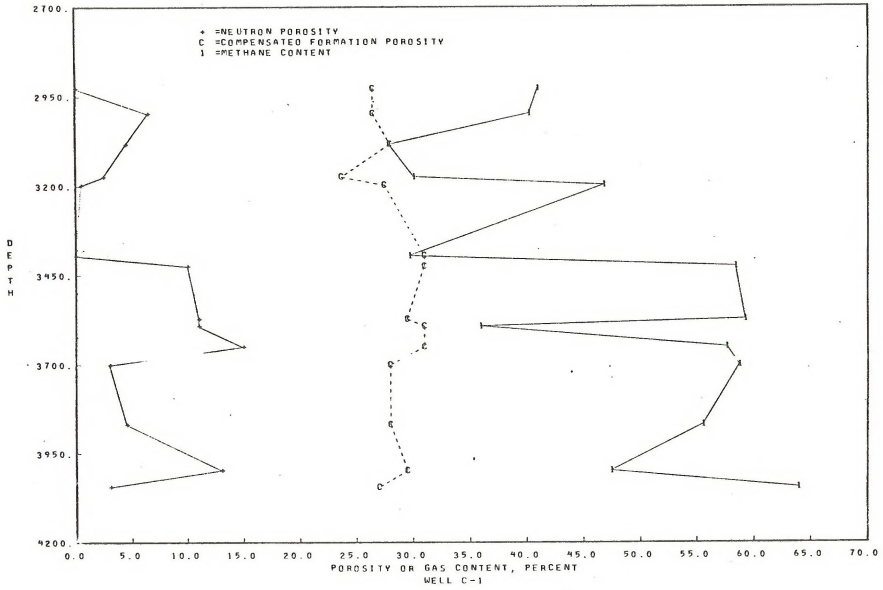


FIGURE 8. SELECTED WELL LOG DATA AND INITIAL METHANE CONTENT

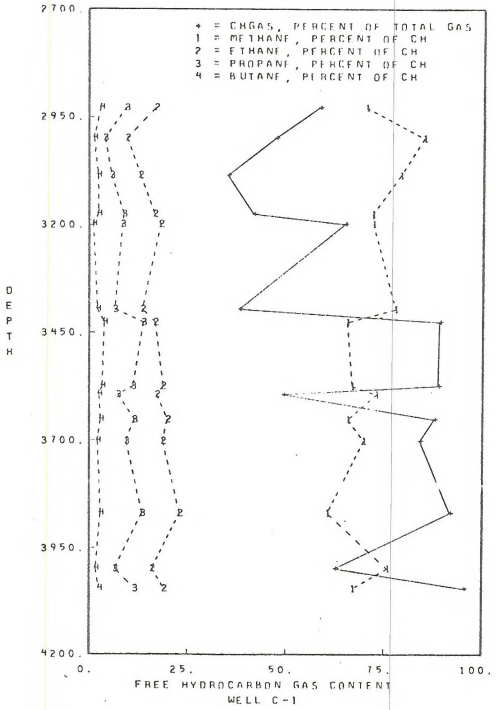


FIGURE 9. TOTAL HYDROCARBON GAS AND RELATIVE AMOUNTS OF VARIOUS HYDROCARBONS

Tasks 3, 4, 5, and 6

After the cans have been punctured and the initial gas release analyses have been completed, the cans are opened for examination of the cores and samples are selected and prepared for physical, chemical, and lithological characterization. Figure 10 is a copy of one of the data sheets used to record the observations and to document sub-sample locations when the cans are opened. A flow sheet indicating the characterization steps is given in Figure 11.

A total of 21 cans have been opened and samples and are at various stages along the characterization chain.

CONCLUSIONS

It is too early in the program and insufficient data are available on any one sample or sets of samples from a given well to draw any conclusions concerning the relationships between shale characteristics and gas release.

Sample No. C 1-3513
Date Opened. 11/9/71.
Engineer E. Y. Harris

Brief mineralogical description: calcite in level A mostly coarse to fine "B" grain, light, "D" mostly coarse dark grain, interbedded as flexible laminae in fine grain matrix containing "C" lens, subhorizontal to A

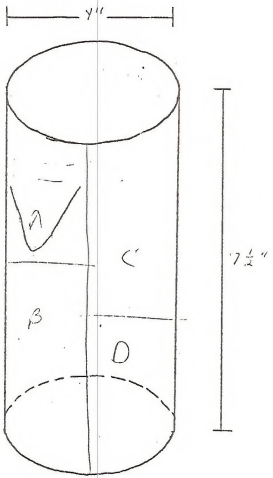
weight of sample + slivers 2457 g
total weight of can + lid 178 g
sample + can weight (Σ) ____ g
initial can weight ____ g
Δ can weight ____ g
Reason ____

Gas build-up detected? Y (N)
Number of pieces in can 4

Samples:

Kinetics 18.1 g D (code)
Bulk ρ 21 (Aplomb) 22 (C) (codes) 23 24 (D) 25 (A-1)
Porosimetry A (A) D (D) (codes)
Permeability B (B) C (C) (codes)
Surface Area D (D) (code)
OES ✓ True ρ ✓
XRD ✓ Excess powder ✓

Can highly rusted



(2) calcite veins (one)

FIGURE 10. OPEN CAN AND SAMPLING DESCRIPTION

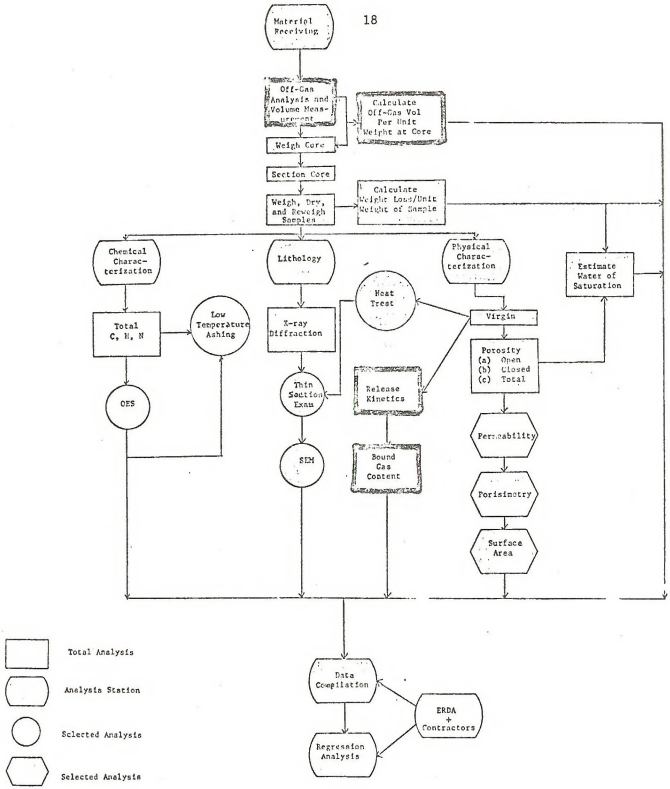


FIGURE 11. CHARACTERIZATION FLOW SHEET